

EFFICIENT SYNTHESIS OF 4, 4'-DIAMINO DIPHENYL METHANES IN A WATER SUSPENSION MEDIUM

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ABSTRACT

The aromatic amines are condensed with formaldehyde to afford the corresponding diamino diphenyl methanes. The method is simple and eco-friendly. The diamino diphenyl methanes are synthesized in high yields using water as a solvent.

Key words : 4, 4'-Diamino diphenylmethanes,

INTRODUCTION

Diamino diphenyl methanes were found to be have various applications like curing agents¹ and chain extenders in polymerization reactions²⁻⁵. Polymerization is an unavoidable process resulting in poor selectivity. These diamino diphenyl methanes are also being used as an intermediate in various reactions. The conventional methods of preparation involve the use of specific mineral acids and alkali at various stages⁶⁻⁹. The use of catalyst under solvent conditions and also in dry media has been already studied⁷⁻¹⁰. The reaction was also been studied under microwave irradiation conditions¹¹⁻¹⁴. In present communication, synthesis of 4, 4'-diamino diphenyl methanes in water solvent without any catalyst has been reported.

EXPERIMENTAL

Melting points were determined in open capillary tubes and are uncorrected. IR spectra were recorded in nujol on Perkin-Elmer 237 spectrophotometer. ¹H NMR was recorded in CDCl₃ on Perkin-Elmer R-32 spectrometer using TMS as an internal standard. (Chemical shift is given in ppm).

Preparation of 2, 2'-dimethyl-4, 4'-diamino diphenyl methane (3)

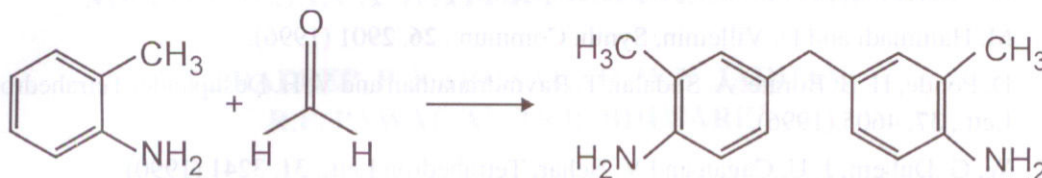
To a slurry of o-toluidine (2 g, 18.66 mmol) in water (50 mL), was added formaldehyde solution (37 %, 0.7 mL, 9.33 mmol) was added slowly with constant stirring till the 2, 2'-dimethyl-4, 4'-diamino diphenyl methane was precipitated out. The solid was filtered, dried and crystallized from ethanol to give (3). ν_{\max} 3487 (N-H) and 1590 cm⁻¹ (C=C). ¹H NMR(δ) : 2.39

Table 1. Physical data of 4, 4'-diamino diphenyl methanes

S.No.	Aniline used	Product	Reaction time in hrs.	Yield (%) ^{a,b}	M.P. (°C)
1			5	78	156
2			0.5	93	178
3			0.5	88	>280
4			2	90	134
5			3	75	>280
6			0.5	79	>280
7			1	93	92
8			6	82	138

^aRefers to isolated yield.^bAll the products exhibited the expected analytical and spectral data.

(s, 3H, CH₃), 3.83 (s, 2H, CH₂), 6.24-6.78 (m, 6H, Ar-H). Similarly all other compounds were also synthesized (Table-1).



Scheme-1

RESULTS AND DISCUSSION

The procedure involves two steps. In first step, the water was mixed with aromatic amines to agitated slurry and in second step, the formaldehyde was added drop wise to the water amine mixture with constant stirring till the reaction was completed.

The method employed is simple, efficient and reproducible. The method is unlike the acid, alkali conventional procedure used. The method developed for this synthesis is without using a catalyst and the conversion was significantly high i.e. 75-93% yields.

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